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X. RELATED PROCEEDINGS APPENDIX

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APPENDIX E

U.S. Patent No. 4,379,169 ("Reggio")

胡木成矿 铁铁矿物 医橡胶物的血病





United States Patent [19]

Reggio et al.

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4,379,169 [11] Apr. 5, 1983 [45]

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|----------------------|---|---|
| [54] | NEW GUM BASE AND CHEWING GUM CONTAINING SAME | 3,974,293 8/1976 Witzel |
| [75] | Inventors: Richard A. Reggio, Yorktown Heights; Ronald P. D'Amelia, Hicksville, both of N.Y.; Dominick R. Friello, Danbury, Conn. | 3,995,064 11/1976 Rhrgoft 426/3 4,064,274 12/1977 Mackay et al. 426/3 4,186,214 1/1980 Crouse et al. 426/3 4,187,320 2/1980 Koch et al. 426/3 4,202,906 5/1980 Ogawa et al. |
| [73] | Assignee: Nabisco Brands, Inc., New York, N.Y. | 4,248,894 2/1981 Mackey et al. 426/3 |
| [21] | Appl. No.: 319,490 | Primary Examiner—Jeanette M. Hunter Attorney, Agent, or Firm—Richard Kornutik |
| [22] | Filed: Nov. 9, 1981 | [57] ABSTRACT |
| [51] [52] [58] | Int. Cl. H23G 3/30 U.S. Cl. 426/3; 426/6 Field of Search 426/3, 4, 5, 6 | A gum base is provided which has excellent film-forming properties and thus is particularly suited for use in a bubble gum. The gum base contains 50 to 80% ester |
| [56] | References Cited | gums and a unique fatty acid or fatty acid ester plasti- |
| | U.S. PATENT DOCUMENTS | cizer, such as glycerol monocleate, and is free of conventional fillers. A bubble gum containing such gum |
| | 3,063,844 11/1962 Meguro et al. | base is also provided. |

426/9

13 Claims, No Drawings



NEW GUM BASE AND CHEWING GUM CONTAINING SAME

FIELD OF THE INVENTION

The present invention which is related to commonly-owned pending application Ser. No. 332,625, filed Dec. 21, 1981, is a novel chewing rum base containing high levels of ester gums, no fillers and a unique plasticizing agent which imparts superior film-forming properties to the gum base, making it especially suited as a bubble gum base. The present invention also relates to chewing gum containing such gum base and to a method for preparing same.

BACKGROUND OF THE INVENTION

Chewing gums available today generally contain a natural rubber gum base, a synthetic rubber gum base or a mixture of natural and synthetic rubber gum bases. In the case of synthetic rubber gum bases, the clastomer 20 usually employed is styrene-butadiene copolymer which is plasticized with glyperol esters of rosin. If a conventional bubble gum base is desired, 25-40% ester gum is usually used as the film-former. Also, appreciable levels of filler, e.g., calcium carbonate, talc, are used to assist in film-forming. Lecithin has also been used in the gum or gum base to soften the extremely firm chew imparted by the use of the ester gums in the gum base. The ester gums, lecithin or other softeners, such as, glycerol monostearate used at required levels to soften 30 the gum base tend to destroy the natural film-forming properties making it undesirable for use as a bubble gum

DESCRIPTION OF THE INVENTION

In accordance with the present invention, a gum base is provided which contains substantial amounts of ester gums yet has highly superior film-forming properties and thus has superior bubble-blowing character, is substantially less tacky than prior art ester gum containing 40 chewing gums and remains soft for prolonged periods. In addition, the gum base of the invention contains no filler and thus may contain mint as well as acid or fruit flavors. The above advantages are achieved in the gum base of the invention by employing from about 50 to 45 about 85% by weight ester gums together with a unique plasticizing agent which includes fatty acids, glycerol ester of fatty acids, polyglycerol esters of fatty acids, or sorbitan or polysorbate esters of fatty acids, and no fillers. Thus, the gum base of the invention will gener- 50 ally comprise one or more natural and/or synthetic elastomers in an amount within the range of from about 0.5 to about 25%, and preferably from about 4 to about 15% by weight of the gum base, ester gum resin in an amount within the range of from about 50 to about 85%, 55 and preferably from about 60 to about 80% by weight of the gum base, plasticizing agent in an amount within the range of from about 1 to about 25%, and preferably from about 5 to about 20% by weight of the gum base, together with softeners in an amount within the range of 60 from about 0 to about 10%, and preferably from about 2 to about 8% by weight of the gum base, and waxes in an amount within the range of from about 1 to about 20%, and preferably from about 3 to about 16% by weight of the gum base.

The unique plasticizing agent whith may be employed in the gum base of the invention includes farry acids, such as oleic acid, lauric acid, lactic acid, isostearic acid, caprylic acid, caprin acid or stripped coco; glycerol esters of fatry acids such as mono, di- or triglycerol esters of any of the fatty acids listed above, with glycerol monocleate being preferred; polyglycerol esters of fatty acids such as any of those listed above, having a hydrophilic/hydrophobic character of HLB 2 to 13; or sorbitan or polysorbate esters of fatty acids such as any of those listed above.

Another of the unique features of the gum base of the invention is the use of the extraordinarily large amounts of ester gums (normally a tackifier) yet the amount of tack in the gum base is less than conventional ester gum type base which contains 25 to 40% ester gum. It is believed that the reduced tackiness in the gum base of the invention is attributed to the maintenance of an ester gum to classomer weight ratio of from about 5:1 to about 8:1 whereas, in convention ester gum containing gum base, such ratio is maintained at or below 4:1. The ester gums which may be employed in the gum base of the invention include any of those normally employed in conventional gum base such as hydrogenated ester gum, that is giveered ester of hydrogenated rosin and/or dimerized ester gum, pentaerythritol ester gum, polymerized ester gum, or ester gum.

In preferred embodiments, the gum base of the invention will contain one or more waxes which serve as texture modifiers and should have a melting point of above about 35° C. The waxes will be present in an amount within the range of from about 1 to about 20%, and preferably from about 3 to about 16% based on the weight of the gum base. Examples of such waxes inolude paraffin wax, microcrystalline wax, carnauba wax, ozokerite wax, oricury wax and the like. Preferred waxes are microcrystalline wax, and paraffin wax employed in combination so that from about 0 to about 15% (based on the weight of the gum base) of the microcrystalline wax is employed with from about 0 to about 15% (based on the weight of the gum base) of the paraffin wax. The waxes are found to reduce the tackiness of the final gum composition without significantly reducing cohesivity thereof.

The gum base of the invention may, but will preferably, contain additional softeners, emulsifiers, and/or lubricants, such as one or more hydrogenated vegetable or animal fats having a melting point above 22° C, in an amount within the range of from about 0 to about 10% and preferably from about 0.5 to about 7% by weight of the gum base. Examples of such softeners include, but are not limited to, glycerol monostearate, lecithin, co-conut oil, fatty acids such as stearic acid, or palmitic acid, partially hydrolyzed polyvinyl esters, or mono, di- and triglycerol esters of fatty acids as described above.

The elastomers which may be present in the gum base of the invention include styrene-butadiene copolymer, isobutylene-isoprene copolymer, polyisobutylene, natural rubber (polyisoprene) as well as other masticatory substances of natural origin, such as rubber latex solids, chicle, crown gum, nispero; rosidinha, jelutong, pendare, perillo, niger gutta, tunu, etc. The elastomer or masticatory substance will be employed in an amount within the range of from about 0.5 to about 25%, preferably from about 4 to about 15% by weight of the gum base.

The following represents preferred gum base formulations in accordance with the present invention.

Ingredient % by Weight Elestomer 4 to 15 (preferred is styreno-butadiene copolymer (24% bound styrene) and/or (48% bound styrene) Ester gum 03 at 08 5 to 20 (preferably glycerol monooleste) (preferably microcrystalline wax end/or paraffin wax) Softeners 0 to 10 (preferably glycerol monosteneste)

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It has been found that, in accordance with the teachings of the present invention, the use of glycerol esters of fatty acids, preferably oleic acid, enhances rather than reduces the film-forming properties of a bubble

It has also been found that where glycerol monooleate is employed as the unique plasticizing agent, an excellent bubble gum base and bubble gum are produced. Glycerol monooleate has been found to be a superior film-forming plasticizing agent for ester gum, 25 far and away better than glycerol monostearate or other conventional gum additives. The film-forming capability of glycerol monopleate increases as its weight percent in the gum base formula increases. Thus, improved bubble blowing capacity is obtained with the use of increasing amounts of glycerol monooleate. In addition, 30 bubble blowing capability is maintained even though the gum base of the invention does not contain conventional fillers, such as calcium carbonate or talc.

The gum base of the invention as described above may be formed by simply mixing the various ingredients 35 thereof until a homogeneous mixture is obtained.

The gum base of the invention may be employed in forming a chewing gum, especially a bubble gum, and in such case the gum base will be present in an amount of within the range of from about 10 to about 40% and 40 preferably from about 15 to about 30% by weight of the chewing gum_

The chewing gum of the invention may be of the sugar-containing or sugarless variety. Examples of sweeteners which may be employed include sugars, for 45 example, monosaccharides of 5 or 6 carbon atoms, such as arabinose, xylose, ribose, glucose, mannose, galactose, fructose, dextrose, or sorbose or mixtures of two or more of the foregoing monosaccharides; disaccharides, for example, sucrose, such as cane or beet augar, 50 lactose, maltose or cellobiose; polysaccharides, such as partially hydrolyzed starch or dextrin, as well as sugar alcohols, such as sorbitol, mannitol, xylitol, or mixtures thereof, as well as hydrogenated starch hydrolysates or isomaltitol, and mixtures of two or more of the above 55 sugars and/or sugar alcohols.

Any of the above sugars may be present in an amount of within the range of from about 0.05 to about 90% and preferably from about 40 to about 85% by weight of the chewing gum. The sugar alcohols, where present, will 60 be employed in an amount of from about 0.05 to about 90% and preferably from about 40 to about 85% by weight of the chewing gum.

The chewing gum of the invention may also contain sugar alcohols an artificial sweetener, such as, for example, aspartame, cyclamate, or a saccharin or other sweetener as set out hereinafter, the artificial sweetener

being present in an amount of from 0 to about 1.5% by weight, and preferably, from about 0.05 to about 0.3% by weight of the chewing gum.

Examples of artificial sweeteners which may be cmplayed herein include sodium, calcium or ammonium saccharin salts, dihydrochalcones, glycyrrhizh, dipotassium glycyrrhizin, glycyrrhizic acid ammonium salt. L-aspartyl-L-phenylalanine methyl ester (aspartame), the sodium, ammonium or calcium salt of 3,4-dihydro-6methyl-1,2,3-oxathiazine-4-one-2,2-dioxide, the potassium salt of 3,4-dihydro-6-methyl-1,2,3-oxathiazine-4-one-2,2-dioxide (Ace-sulfame-K), as well as Stevia rebaudiana (Stevioside), Richardella dulcifica (Miracle Berry), Dioscoreophyllum cumminsli (Serendipity Berry), cyclamate salts, and the like, or mixtures of any two or more of the above.

The chewing gum of the invention may include Ilavoring, such as sour or fruit flavoring or non-acid or mint flavoring in an amount ranging from about 0.5 to about 2% by weight of the final chewing gum product. The flavoring may comprise synthetic flavors and oils derived from plants, leaves, flowers, fruit, etc. Representative fruit flavor adjuncts include acids, such as adipic, citric, malic, succinic and fumaric acid, and citrus oils, such as lemon oil, orange oil, lime oil, grapefruit oil, and fruit essences, such as apple essence, pear essence, peach essence, strawberry essence, apricot essence, raspberry essence, cherry essence, plum essence, pineapple essence, as well as the following essential oils: peppermint oil, spearmint oil, mixtures of peppermint oil and spearmint oil, clove oil, bay oil, anise oil, eucalyptus oil, thyme oil, cedar leaf oil, cinnamon oil, oil of nutmeg, oil of sage, oil of bitter almonds, cassia oil, and methylsalicylate (oil of wintergreen). Various synthetic flavors, such as mixed fruit, may also be incorporated in the chewable gum base with or without conventional preservatives.

The above-described chewing gums containing the unique gum base of the invention may be prepared employing conventional processing techniques.

The following Examples represent preferred embodiments of the present invention.

EXAMPLE 1

A bubble gum base of the following formulation was prepaed as described below.

| | Gum Base Ingredients | % by Weight |
|----|--|-------------|
| 7. | Styrene-butadiene elastomer | , |
| | (a) 24% bound styrene | 3 |
| • | (b) 48% bound styrene | 7 |
| | Ester gam (glycerol ester of modified rosin) | ìo |
| | Ester gum (glycerol ester of hydrogenated rosin) | 60 |
| | Glycerol monocleate | . 3 |
| | Glycerol monosteamte | . 2 |
| • | Triglyceride | 2 |
| | Paraltin wax | 6 |
| | Microcrystalline wax | |
| • | | 100% |

The elastomer and ester gums were mixed in a sigma in lieu of or in addition to any of the above sugars or 65 blade mixer until homogeneous. Thereafter, the waxes were added with mixing followed by the remaining ingredients. Mixing was continued until a homogeneous mass was obtained.



The above gum base of the invention chews well, has reduced tackiness and has good bubble-blowing properties.

In addition, since it is free of CaCO1, the gum base may be used with acid flavors and/or acid sweeteners 5 such as aspartame and free acid form of saccharin. Also, the addition of 70% ester gum surprisingly does not adversely affect the abhesive properties of the base, and, in fact, makes the base less tacky.

EXAMPLE 2

A bubble gum base of the following formulation was prepared as described below.

| Gum Base Ingredient | | | % by Weight |
|----------------------------|-----------|---|-------------|
| Styrene-butadiene elastion | | : | |
| (30:70 mix of 24% bound | | | |
| and 48% bound styrene of | raterral) | | 10 |
| Ester gum | : | | 10 . |
| Ester gum | · | | 67. |
| Glycerol monocleate | • | • | \$ |
| Microcrystalline wax | | | 7 . |
| Paraffin wax | 1 | | 6 |

The elastomer and ester gums were mixed in a sigma 25 blade mixer until homogeneous. Thereafter, the waxes were added with mixing followed by the glycerol monooleate. Mixing was continued until a homogeneous mass was obtained.

The above gum base of the invention chews well, has 30 reduced tackiness and has very good bubble-blowing

In addition, since it is free of CaCO3, the gum base may be used with acid flavors and/or acid sweeteners such as aspartame and free acid form of saccharin. Also, the addition of 72% ester gum surprisingly does not adversely affect the abhesive properties of the base, and, in fact, makes the base less tacky.

EXAMPLE 3

A bubble gum base of the following formulation was prepared as described in Example 2.

| Guro Basc Ingrediem | % by Weight | - 4 |
|------------------------------------|-------------|----------------|
| Styrene-butadiene elastomer (30:70 | | - |
| of 24% and 48% bound styrene) | 1D | |
| Ester gum | 10 | |
| Eater gum | 60 | |
| Olycznel monopleate | 10 | - |
| Microcrystalline wax | . 5 | 2 |
| Parallin was | ž | |

The above gum base of the invention chews well, has reduced tackiness and has excellent bubble-blowing 55 reduced tackiness, and has very good bubble-blowing properties.

In addition, since it is free of CaCO3, the gum base may be used with acid flavors and/or acid sweeteners such as aspartame and free acid form of saccharin. Also, the addition of 70% ester gum surprisingly does not so adversely affect the abhesive properties of the base, and, in fact, makes the base less tacky.

In a control run, to demonstrate the superiority of glycerol monooleate over glycerol monostearate in. increasing film-forming capability of ester gum, the 65 following was prepared as described in Example 2, except that glycerol monostearate was used in place of glycerol monooleate.

| | | | _ |
|------------------------------------|---|--|--|
| Control Ran A | | | |
| and the tifficular | _ | NO DY WEIGHT | _ |
| Styrene-butadiene elestomer (30-70 | | | |
| mix of 24% and 48% hound growne) | • | | |
| | | | |
| | | . 10 | |
| Ester gum | | 60 | |
| Olycerol monostearate | : | | |
| | | | |
| Pa-On | | · . | |
| Larging wit | | 5. | |
| | Control Ran A Gum Base Ingredient Styrene-butadiene elastomer (30:70 mix of 24% and 48% bound styrene) Ester gum Exter gum Cityeerol monostearate Microcrystalline wax Paraffin wax | Gum Base Ingredient Styrene-butadiene elastomer (30:70 mix of 24% and 48% bound styrene) Ester gum Exter gum Olyverol monostearate Microcrystalline wax | Gum Base Ingredlent % by Weight Styrene-butadiene elastomer (30:70 mix of 24% and 48% bound styrene) 10 Ester gum 10 Ester gum 60 Olyverol monostearate 10 Microcrystalline wax 5 |

The gum base so-prepared is found to be a poor bubble gum, with poor bubble blowing capability, thereby clearly evidencing superiorty of glycerol monooleste . 15 over glycerol monostearate.

EXAMPLE 4 ·

A bubble gum base of the following formulation was prepared as described in Example 2.

| Gum Base Ingredient | | % by Weight | |
|---|---|----------------------------|--|
| Styrene-buttediene eles mix of 24% and 48% Ester gum Ester gum | | 10 · 10 · 10 · 160 · . · . | |
| Glycerol monooleate Microcrystalline wax | • | 13 | |

The above gum base of the invention chews well, has reduced tackiness, and has excellent bubble-blowing properties. The gum base is initially very soft and remains soft over extended periods covering several weeks.

In addition, since it is free of CaCO3, the gum base may be used with acid flavors and/or acid sweeteners such as aspartame and free acid form of saccharin. Also, the addition of 70% ester gum surprisingly does not adversely affect the abhesive properties of the base, and, in fact, makes the base less tacky. ΔN

EXAMPLE 5 ..

A bubble gum base of the following formulation was prepared as described in Example 2.

| Gun Base Ingredient | % by Weight |
|-----------------------------|-------------|
| Styrene-butadiene elastomer | |
| (48% bound styrene) | 10 |
| Exter gum | 10 |
| Enter gum | . 63 |
| Oleio seid | . 10 |
| Microcrystallins wax | . 5 |

The above gum base of the invention chews well, has properties. The gum base is initially very soft and remains soft over extended periods covering several weeks.

In addition, since it is free of CaCO3, the gum base may be used with acid flavors and/or acid sweeteners such as aspartance and free acid form of saccharin. Also, the addition of 75% ester guin surprisingly does not adversely affect the abhesive properties of the base, and, in fact, makes the base less tacky.

EXAMPLE 6

A bubble gum base of the following formulation was prepared as described in Brample 2.

| Gam Base Ingredient | % by Weight | | |
|--|----------------|--|--|
| Styrone-butadiene elestomer (48% bound styrene) Ester gum Ester gum | 01 01 28 | | |
| Polyglyceral ester of aleic acid Microcrystalline wax | 10 5 | | |

The above gum base of the invention chaws well, has reduced tackiness, and has excellent bubble-blowing properties. The gum base is initially very soft and remains soft over extended periods covering several weeks.

In addition, since it is free of CaCO₃, the gum base may be used with acid flavors and/or acid sweeteners such as aspartame and free acid form of saccharin. Also, the addition of 75% ester gum surprisingly does not adversely affect the abhasive properties of the base, and, ²⁰ in fact, makes the base less tacky.

EXAMPLE 7

A bubble gum base of the following formulation was prepared as described in Example 2.

| Gum Bass Ingredient | % by Weight |
|-----------------------------|-------------|
| Styrene-butadiene elastomer | |
| 48% bound styrene) | 10 |
| ster gum | 10 |
| ister gun | 65 |
| Tween 85 | 10 |
| Microcrystalline wex | 5 |

The above gum base of the invention chews well, has reduced tackiness, and has good bubble-blowing properties. The gum base is initially very sot and remains soft over extended periods covering several weeks.

In addition, since it is free of CaCO₃, the gum base may be used with acid flavors and/or acid sweeteners such as aspartame and free acid form of saccharin. Also, the addition of 75% ester gum surprisingly does not adversely affect the abhesive properties of the base, and, 45 in fact, makes the base less tacky.

In a control run to further demonstrate the superiority of glycerol monooleate over glycerol monostearate in increasing film-forming capability of estex gum, the following gum base was prepared as described in Control Run A.

| . Control Run B | | _ |
|---------------------------------|-------------|-----|
| Gum Base Ingredient : | % by Weight | . 5 |
| Styreno-butedisne elastomer | ;· · · | _ |
| (30:70 mix of 24% and 48% bound | | |
| styrene) | . 10 | |
| Eater gum | 10 | ; . |
| Ester gum | 60 | |
| Glycerol monostearete | 15 | 60 |
| Microcrystalline wax | 5 | |

The gum base produced is found to be initially hard and gets harder on standing, and is very difficult to 65 blow bubbles with. Thus, it is again seen that glycerol monostearate is not an effective film-forming agent for ester gums.

EXAMPLE 8

A bubble gum having the following composition was prepared as described below:

| Ingredient | % by Weight of the Chewing Gum |
|------------------------|-----------------------------------|
| Gum base (as described | |
| in Ex. 1) | 22 · |
| Sugar polyerized | · <u>5</u> 2 |
| Com syrup 43° Be | 23 |
| Softeners | 15 |
| Flavor | 1.0 |
| Color | 0.05 |

The gum base was melted (temperature 250° P.) and placed in a standard dough mixer kettle equipped with sigma blades and cooled to 180° F. The corn syrup, softeners and color were added with mixing over a 5 minute period, thereafter the pulverized sugar and flavors were added according to conventional chewing gum practice and mixed for 5 minutes. The gum was discharged from the kettle and was rolled or extruded and cut into sticks or cubes.

The resulting chewing gum product is found to have a good chew and has improved bubble blowing properties.

EXAMPLE 9

A bubble gum having the following composition was prepared as described below:

| Ingredient | % by Weight of the Chewing Gum | • |
|--------------------------|-----------------------------------|---|
| Gum base (as described | | |
| in Ex. 2) | 24 | • |
| Sugar pulverized | 61 | : |
| Com syrup, high fructose | 14 | • |
| Flevor | 1 | |

The gum base was melted (temperature 250° F.) and placed in a standard dough mixer kettle equipped with sigma blades and cooled to 180° F. The corn syrup was added with mixing over a 5 minute period thereafter the pulverized sugar and flavors were added according to conventional chewing gum practice and mixed for 5 minutes. The gum was discharged from the kettle and was rolled or extruded and cut into sticks or cubes.

The resulting chewing gum product is found to have a good chew and has very good bubble blowing properties.

EXAMPLE 10

A sugarless bubble gum in accordance with the present invention and having the following composition was prepared as described below:

| Ingradient | % by Weight of the Chewing Gum | |
|------------------------|-----------------------------------|--|
| Oun base (as described | | |
| in Ex. 3) | 26 | |
| " Sorbitol | 49.4 | |
| Mappitol | 1.5 | |
| Sorbitol solution . | · 19.5 | |
| Flavor | 1.5 | |
| Sodhum saccharin | 0.1 | |

The gum base was melted (temperature 250° R.) and placed in a standard dough mixer kettle equipped with sigma blades and cooled to 180° R. The mannitol and sorbitol powder were added with mixing over a 5 minute period; thereafter the flavor, sorbitol solution and sodium saccharin were added according to conventional chewing gum practice and mixed for 5 minutes. The gum was discharged from the kettle and was rolled or extruded and cut into sticks or cubes.

The resulting chewing gum product is found to have a good chew, a pleasant sweet taste and has excellent bubble blowing properties.

EXAMPLE 11

A sugarless bubble gum in accordance with the present invention and having the following composition was prepared as described below:

| | <u>:</u> | • |
|-------------------------|------------------------------------|---|
| Ingredient | % by Weight of the Chewing Guro | |
| Outs base (as described | : | |
| in Ex. 4) | . 24 | |
| Sorbital powder | . as | |
| Mannitol | 5 | |
| Plavor | 1.5 | |
| Water | 6 | |
| | | |

The gum base was melted (temperature 250° F.) and placed in a standard dough mixer ketile equipped with 30 sigma blades and cooled to 180° P. The powdered sorbitol and mannitol were added with mixing over a 5 minute period; thereafter the flavor and water were added according to conventional chewing gum practice and mixed for 5 minutes. The gum was discharged from the 35 kettle and was rolled or extruded and cut into sticks or cubes.

The resulting chewing gum product is found to have a good chew and has excellent bubble blowing properties and reduced tackiness.

EXAMPLE 12

A bubble gum having the following composition was prepared as described below:

| Ingredient | % by Weight of the Chewing Gum | |
|------------------------|-----------------------------------|--|
| Gum base (as described | | |
| in Ex. 5) | 24 | |
| Sugar pulverized | 52 | |
| Corn syrup 43° Re | 21 | |
| Softeners | | |
| Flavor | 1.93 | |
| | i I | |
| Color | : 0.05 | |

The gum base was melted (temperature 250° F.) and placed in a standard dough mixer kettle equipped with sigma blades and cooled to 180° F. The corn syrup, softeners and color were added with mixing over a 5 minute period; thereafter the pulverized sugar and flavors were added according to conventional chewing gum practice and mixed for 5 minutes. The gum was discharged from the kettle and was rolled or extruded and cut into sticks or cubes.

The resulting chewing gum product is found to have a good chew and has very good bubble blowing properties.

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EXAMPLE 13

A bubble gum having the following composition was prepared as described below:

| ٠. | Ingredient | the Chewing Gum | |
|----|-----------------------------------|-----------------|--|
| | Gum base (as described | | |
| | h Ex 6) | . 22 | |
| | Sugar pulverized | 52.5 | |
| | Sogar pulverized Com syrup 43° Be | | |
| | Safteners | 0.75 | |
| | . Flavor . | • | |
| | Color . | 1.2 | |
| | Citrio acid | 0.05 1.5 | |

The gum base was melted (temperature 250° F.) and placed in a standard dough mixer kettle equipped with sigma blades and cooled to 180° F. The corn syrup, softeners and color were added with mixing over a 5 minute period; thereafter the pulverized sugar and flavors were added according to conventional chewing gum practice and mixed for 5 minutes. The gum was discharged from the kettle and was rolled or extruded and cut into sticks or cubes.

The resulting chewing gum product is found to have a good chew, tart flavor, has excellent bubble blowing properties, and exhibits reduced tackiness.

EXAMPLE 14.

A bubble gum having the following composition was prepared as described below:

| Ingredient | % by Weight of the Chewing Gum | |
|------------------------|-----------------------------------|--|
| Gum base (as described | | |
| so Ex. 7) | 22 | |
| Sugar pulverized | \$2.95 | |
| Corn syrup 43' Be | . 23 | |
| Softeners | . 7 | |
| Flavor | ; | |
| Color | 0.03 | |

The gum base was melted (temperature 250° F.) and placed in a standard dough mixer kettle equipped with sigma blades and cooled to 180° F. The corn syrup, softeners and color were added with mixing over a 5 minute period; thereafter the pulverized sugar and flavors were added according to conventional chowing gum practice and mixed for 5 minutes. The gum was discharged from the kettle and was rolled or extruded and cut into sticks or cubes.

The resulting chewing gum product is found to have a good chew and has good bubble blowing properties. What is claimed is:

1. A gum base which has excellent film-forming capability, comprising from about 0.5 to about 25% by weight of an elastomer, from about 50 to about 85% by weight of one or more ester gums, and from about 1 to about 25% by weight of a plasticizer selected from the group consisting of acids selected from the group consisting of oleic acid, lauric acid, lactic acid, isostearic acid, caprylic acid and capric acid, esters of said acids, mono-, di- and triglycerol esters of said acids, polyglycerol esters of said acids having a hydrophobic hydrophilic character of HLB 2 to 13, sorbitan or polysorbate ester of said acids, atripped coco, and mixtures thereof,

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said gum base being substantially free of inorganic fill-

2. The gum base as defined in claim 1 wherein said plasticizer is glycerol monooleate.

3. The gum base as defined in claim 2 wherein said 5 glycerol monooleste is present in an amount within the range of from about 3 to about 20% by weight of said

4. The gum base as defined in claim 1 wherein said polyglycerol ester of a fatty acid is present in an amount within the range from about 3 to about 20% by weight of said gum base.

5. The gum base as defined in claim 1 wherein said ester gum is present in an amount within the range of 15 gum base is a bubble gum base. from about 50 to about 80% by weight of said gum base.

6. The gum base as defined in claim 1 wherein said ester gum is glycerol ester of hydrogenated rosin, dimerized ester gum, pentaerythritol ester gum, polymerized ester gum, ester gum or mixtures thereof.

7. The gum base as defined in claim 1 wherein said clastomer is a styrene-butadiene copolymer, polyisobutylene, isobutylene-isoprene copolymer, or natural rub-

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8. The gum base as defined in claim 1 further including one or more waxes.

9. The gum base as defined in claim 8 wherein said waxes are present in an amount within the range of from about 1 to about 20% by weight of said gum base.

10. The gum base as defined in claim 8 further including one or more additional softeners present in an amount within the range of from about 0 to about 10% by weight of said gum base.

11. The gum base as defined in claim 1 wherein said

12. A chewing gum containing a gum base as defined in claim 1.

13. A bubble gum containing gum base as defined in claim 1.

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APPENDIX F

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- [72] Inventor: François Boudy
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[57] ABSTRACT

The invention relates to a process for preparing a gum base concentrate with a high polymer content of the type including high molecular weight elastomers, plasticizers and mineral fillers.

This process is characterized by the fact that the high molecular weight elastomers are ground into particles, these particles are mixed in the desired proportions and plasticized; the resulting mixture and the mineral fillers are introduced separately in the desired proportions in a twin-screw extruder 6 with sleeves 0, 1', 2', 3',

120°C or the elastomer mixture does not exceed than about one minute and thirty seconds, and whose screw speed is less than about 120 rpm, and the concentrate exiting the extruder is recovered.

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GUM BASE CONCENTRATES WITH HIGH POLYMER CONTENT, THEIR PREPARATION PROCESS AND THEIR USE IN CHEVING GUM PRODUCTION

The present invention relates to gum base concentrates with high polymer content which can be used in the preparation of gum base for chewing gums.

The invention also relates to the preparation of such concentrates and their use in the production of cheving gums.

It is known that gum bases normally include high molecular weight polymers, mineral fillers and plasticizers. High molecular weight polymers include, but are not limited to, isoprene-isobutylene copolymers, styrene, butadiene and polyisobutylene, while low molecular weight polymers include polyisobutylene and polyvinyl acetate. Plasticizers include resins of rosin and waxes.

Gum bases can be prepared directly by the chewing gum manufacturers.

They can also be produced separately and be sold as semi-finished products.

They are usually produced in two stages.

In a first stage, a premix based on polymers, plasticizers and mineral fillers is prepared. This premix is then transferred into a mixer, where the rest of the usual components of the gum base are added. This second stage does not concern the present invention, and we will discuss in the following only the production and formulation of the premixes.

The premixes can be prepared by dissolving the main polymer at a high temperature in the other materials. This dissolving operation lasts about 3-6 hours and is performed in an open mixer with moveable blades called a "pug mill," in which the mixture is held between 120 and 150°C throughout the operation. This technique, however, has the drawback that the polymer undergoes degradations in the elastic chains due to the temperature at which

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the dissolving is performed and due to the duration of a cycle. As a result, the product obtained has mediocre elasticity, whereas good elasticity is an important characteristic of a satisfactory premix.

The premix of the base polymer and the other materials can also be performed at constant volume and under pressure in a high-powered shearing mixer known as a "pug mill." The duration of a mixing cycle is 20-30 minutes, and the product undergoes considerable mechanical and thermal stresses.

This process, like the one described above, thus involves degradation of the elastic properties of the polymer. Furthermore, such a process cannot be applied to the preparation of tacky or viscous products, and it requires the resulting mixture to have a certain consistency.

Finally, the premix can be conducted by co-extruding the different components, but this process, which combines the effects of temperature, pressure and shearing, also involves degradation of the elastic properties of the polymer.

Regardless of the technique used to prepare these premixes, they never contained more than about 50% polymer by weight.

The present invention intends to remedy the drawbacks of the above-described prior art.

One purpose of the present invention is therefore to propose a process for preparing a concentrate with a high polymer content that can attain and exceed 85% by weight, which does not substantially alter the elastic properties of these polymers.

Another purpose of the invention is to propose a process that can be used without restrictions for preparing the concentrate with a high content of polymers, even viscous or tacky ones.

Another purpose of the invention is a concentrate whose polymer content can attain and exceed 85% by weight.

The invention also intends to propose a concentrate of this type that exhibits satisfactory elasticity for preparing chewing gums.

Finally, the invention aims to propose a concentrate such that industrialists can easily apply it to the preparation of their own products by adding to it the additives they normally use in the necessary proportions.

For this purpose, the object of the invention is a process for preparing a gum base concentrate with a high polymer content of the type including high molecular weight elastomers, plasticizers and mineral fillers, characterized by the fact that high molecular weight elastomers are ground to the form of particles; these particles and plasticizers are mixed in the desired proportions; the resulting mixture and the mineral fillers are introduced separately in the desired proportions in a twin-screw extruder with sleeves, in which the temperature does not exceed 120°C, in which the elastomer mixture does not spend more than about 1 min 30 sec, and whose screw speed is less than about 120 rpm, and the concentrate exiting the extruder is recovered.

In this definition of the process according to the invention, in the rest of the description and in the claims that follow, the term high molecular weight elastomers refers to elastomers having a numerical molecular weight of at least 220,000 and a molecular weight by weight of at least 450,000.

Advantageously, the mixture of high molecular weight elastomer and plasticizers will be introduced into the sleeve arranged furthest upstream in the twin-screw extruder, while the mineral fillers are introduced downstream, preferably in two different sleeves of the extruder to enhance their

distribution in the finished product. The particles of ground elastomer may have a size on the order of 5 mm.

In the event tacky materials such as low molecular weight elastomers must be present in the concentration thus prepared, they are introduced in the molten state into the twin-screw extruder downstream from the mixture of high molecular weight elastomers and plasticizers and upstream from the mineral fillers. By low molecular weight elastomers we mean elastomers having a molecular weight by number less than 4,000 and a molecular weight by weight less than 150,000.

The process in accordance with the invention therefore aims to avoid exposing the elastic chains of the elastomer to excessive temperature and shear stresses or subjecting them to such stresses for too long.

An embodiment of this process will be described in the following in greater detail as a non limiting example with reference to the attendant drawings. In these drawings:

Figure 1 is a schematic view of the apparatus used in this embodiment of the process;

Figures 2a. 2b and 2c are diagrams illustrating the conditions of operation of the five-sleeve extruder of this apparatus.

As we see in figure 1, the high molecular weight elastomer is first ground into particles of the same size of five millimeters in an impact disc mill 1. The ground product is then mixed in a ribbon mixer 2 with plasticizers (resins of rosin, waxes, etc) fed through line 3, and the resulting mixture is transferred into a vibrating weight metering hopper 4.

The mineral fillers to be added to the product (talc and calcium carbonate) are loaded into two vibrating weight metering hoppers 5.

These different metering hoppers then introduce the materials they contained in specific proportions into a twin-screw extruder 6, which in this case, is a "five-sleeve" extruder (recall that, in the usual classification of extruders, the input sleeve, or sleeve 0, is not taken into account; in the present installation, the extruder thus actually includes six sleeves 0, 1', 2', 3', 4' and 5'). More precisely, after preheating twin-screw extruder 6, while the screws are driven by the motor, the premix consisting of high molecular weight elastomer and plasticizers is fed into sleeve 0, or the input sleeve of the extruder, while the mineral fillers are introduced by the two metering hoppers 5 in two different sleeves, for example, sleeves 0 and 2', of the twin-screw extruder to improve the homogeneity of the finished product.

If the concentrate being prepared must contain low molecular weight elastomers, the latter are fed in the molten state into a tank 8, heated to a temperature of 100-120°C by oil circulating in a closed circuit 9 from a boiler 10. These elastomers are then injected by a gear pump 11 into sleeve 1'.

The entire assembly may, naturally, operate continuously.

The screws of extruder 6 may rotate in the same or different directions.

For a given flow rate, their rotational speed will be chosen such as to minimize degradation of the polymer while insuring satisfactory entry of the materials into the machine.

At the rate of 120 kg/hr, using a five-sleeve extruder whose screws have a diameter of 64 mm and a length equal to 29 times the diameter (extruder type

known as 29 D), the rotational speed of the screws may range from 80 to 100 rpm.

For a five-sleeve extruder in each case, the following table gives the temperatures for each of the sleeves.

| Sleeve | Temperature |
|------------------|-------------|
| 0 | • |
| 1. | 12°C |
| .2 * | 60°C |
| 3· | 60.C |
| <u>.</u> | 80°C |
| 5, | 80.C |
| Extrusion nozzle | 80°C |
| | . 70°C |

After a residence time of 1 min 30 sec in the twin-screw extruder, the concentrate with high polymer content exits the extrusion nozzle at a temperature in the range of 100-120°C.

Figures 2a, 2b and 2c, which must be considered as arranged end-to-end, illustrate the treatments undergone in this case by the materials mixed in the five sleeves of the extruder. The abbreviations used in these diagrams have the following definitions:

TSF: transfer,

LM: low-shear mixing,

HM: high-shear mixing,

CM: counterthread mixing.

By applying the above conditions, the apparatus we have just described makes it possible to produce concentrates with high polymer contents having satisfactory elasticity for the preparation of chewing gums.

The concentrates thus prepared may thus have, depending on the intended use, a composition within the following composition range, in weight percent:

5 to 10%

- high molecular weight elastomers: 30 to 85%
- low molecular weight elastomers: 0 to 5%
- rosin resin: 8 to 10%
- wax-type plasticizer: 2 to 10%
- mineral fillers: 5 to 10%

A gum base concentrate intended for the preparation of chewing gum tablets will preferably have the following composition in weight percent:

- elastomers: 85x
- mineral fillers: 18x
- plasticizers: 2x

The high molecular weight elastomers used in this case may have molecular weights in number Mn and in weight Mw which vary depending on the rotary speed of the five-sleeve twin-screw extruder's screw.

These weights will be, for example, as follows:

| | Mo | Mw |
|------------------------------------|---------|---------|
| — Continuous production at 80 rpm | 135 900 | 208 800 |
| - Continuous production at 100 rpm | | 200 000 |
| - Continuous production at 120 rpm | 128 800 | 196 400 |

For a concentrate intended for the preparation of bubble gum, a preferred composition will be as follows in weight percent:

elastomers: 32%
mineral fillers: 52%
plasticizers: 16%

The high molecular weight elastomers used in this case may have the following characteristics as a function of the speed of the five-sleeve twinscrew extruder screw:

| | Hn | Mu | |
|------------------------------------|--------|---------|---|
| - Continuous production at 80 rpm | 86 500 | 459 700 | |
| — Continuous production at 100 rpm | 79 700 | 609 000 | ٠ |

In the concentrates thus prepared, the initial elastic properties of the elastomers are not altered by the preparation process and are practically unchanged in the finished product.

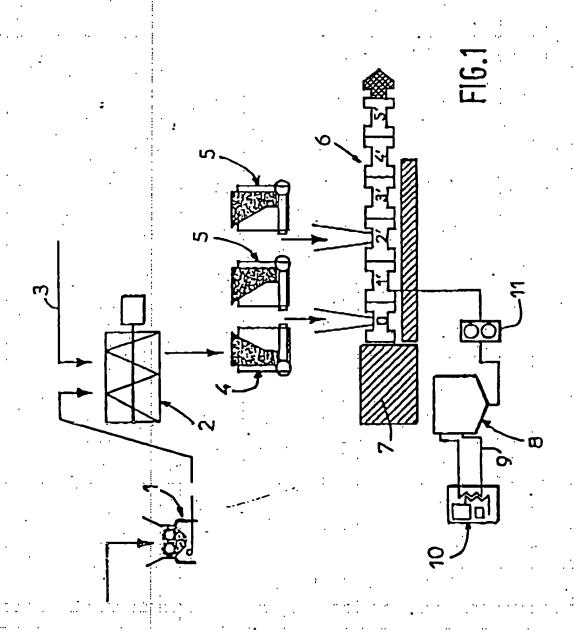
Claims

- 1. Process for preparing a gum base concentrate with a high polymer content of the type including high molecular weight elastomers, plasticizers and mineral fillers, characterized by the fact that the high molecular weight elastomers are ground into particles; these particles and the plasticizers are mixed in the desired proportions; the resulting mixture and the mineral fillers are introduced separately in the desired proportions into a twin-screw extruder (6) with sleeves (0, 1', 2', 3', 4', 5') in which the temperature does not exceed 120°C, where the elastomer mixture does not spend more than about 1 minute and 30 seconds, and whose screw speed is less than about 120 rpm, and the concentrate exiting the extruder is recovered.
- 2. Process according to claim 1, characterized by the fact that the high molecular weight elastomers are ground into particles having a size on the order of 5 mm.
- 3. Process according to one of claims 1 and 2, characterized by the fact that the mixture of high molecular weight elastomers and plasticizers is introduced into the sleeve (0) arranged furthest upstream in the twin-screw extruder (6), while the mineral fillers are introduced downstream from this mixture.
- 4. Process according to claim 3, characterized by the fact that the mineral fillers are introduced in two different sleeves (0, 2') of the twinscrew extruder (6).
- 5. Process according to one of claims 1 through 4, characterized by the fact that low molecular weight elastomers are introduced in the molten state

in the twin-screw extruder (6) downstream from the mixture of high molecular weight elastomers.

- 6. Process according to one of claims 1 through 5, characterized by the fact that the twin-screw extruder (6) is an extruder with five sleeves (0, 1', 2', 3', 4', 5').
- 7. Gum base concentrate prepared by a process according to one of claims 1 through 6.
- 8. Gum base concentrate characterized by the fact that it includes at least 30%, preferably 85% polymers by weight.
- 9. Use of a gum base concentrate according to one of claims 1 through 8 for preparing chewing gum.

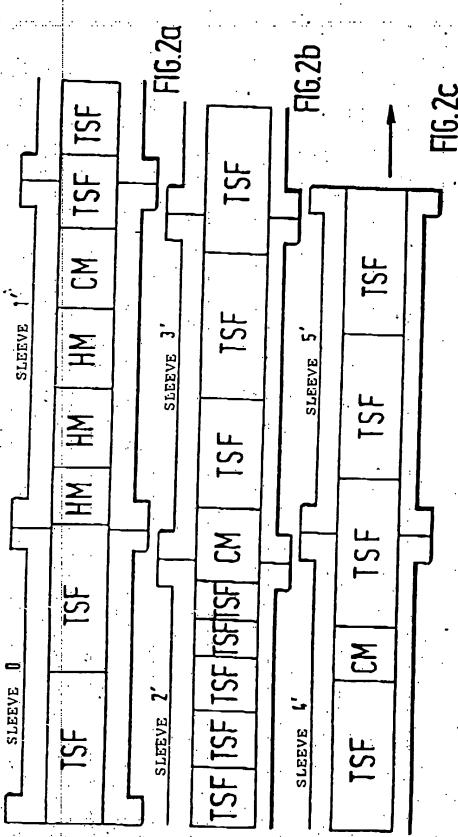
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